

# AMS Measurements of Actinides and technetium at LLNL

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# AMS measurements of actinides and technetium at LLNL<sup>†,§</sup>

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## 1. Introduction

The Center for Accelerator Mass Spectrometry (CAMS) and the Health and Ecological Assessment Division (HEA) at Lawrence Livermore National Laboratory (LLNL) are developing methods for the measurement of technetium and plutonium as part of a long-term effort to expand our accelerator mass spectrometry (AMS) capabilities to isotopes throughout the periodic table. AMS provides ultratrace sensitivity for these nuclides with high rejection of interferences and low susceptibility to matrix components. These advantages will lead to reduced demands on the sample preparation chemistry, high throughput, and rapid turn around of results. Ultimately these will result in larger, more reliable, and more complete data sets.

We have begun measurements of <sup>99</sup>Tc and Pu isotopes in environmental samples, including IAEA reference materials. The following sections contain a summary of our sample preparation and AMS measurement protocols, and the results of some recent measurements. It should be noted that the results presented here are mostly of a preliminary nature, and are meant only to demonstrate the current status of the methods.

## 2. Technetium-99 measurements

### *Technical Issues*

Technetium-99 AMS presents unique technical challenges which are not encountered for the other radionuclides more typically measured by AMS. These challenges relate to the lack of a stable technetium isotope (McAninch and Proctor 1995; McAninch, Marchetti et al. 1998). In AMS measurements of other long-lived radionuclides, stable isotopes of the element of interest implicitly fill several important roles such as chemical carrier, sample matrix, internal yield monitor, and instrument normalization. In the case of <sup>99</sup>Tc, these roles must be filled by some combination of other elements and other (radioactive) technetium isotopes. In our work, we use a combination of niobium, as sample matrix and instrument normalization, and <sup>95m</sup>Tc, as chemical yield monitor, to fill these roles. While these solutions allow quantitative measurement of <sup>99</sup>Tc at femtogram levels, they also represent a series of compromises which at present limit the instrumental precision to 15-25%.

A further challenge in mass spectrometric measurements of <sup>99</sup>Tc is background from the isobaric interference <sup>99</sup>Ru. AMS provides a significant advantage in this regard in that the high energy of the resulting ions allows the use of nuclear-physics type particle identification for the post-spectrometer rejection of this and other interferences. For the energies attainable with the CAMS spectrometer, a rejection of <sup>99</sup>Ru by up to two orders of magnitude is obtained. The

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resulting sensitivity for environmental samples is ~10-20 fg, depending on the chemical recovery of technetium and the level of ruthenium remaining.

### ***Sample Preparation***

Efforts to this point have focused on aqueous samples or simple leachates of soils. Aqueous samples or leachates are reduced in volume to ~100 mL (small samples are diluted to ~100 mL) and boiled with peroxide to fully oxidize technetium and to destroy organics. For some samples, persulfate is also added generate a more aggressive oxidation. Calcium or barium are added for some samples to precipitate major matrix components. Samples are then filtered at 0.2  $\mu\text{m}$  and loaded onto a column containing 2 mL of a technetium-specific resin (Eichrom TEVA-Spec<sup>TM</sup>). Following rinsing with 0.1 and 1 M nitric acid, Tc is eluted with 15 mL of 8 M nitric into a teflon vial.

Niobium is added to the eluent in a dissolved form. A weighed amount of peroxoniobium solution (2 mg total Nb in concentrated peroxide/nitric acid solution) is added to the sample. The vial is capped with a flow-through lid and the samples are dried under a dry nitrogen flow at low temperature on a hot plate. Once dried, the niobium oxide pellet is transferred to a small borosilicate test tube and baked at 400 C for ~1 hr. The pellet is then crushed to improve sample homogeneity, and pressed into a standard LLNL aluminum AMS sample holder.

Technetium recovery is monitored using  $^{95\text{m}}\text{Tc}$  as a tracer. The  $^{95\text{m}}\text{Tc}$  stock used at CAMS to date was extracted from a spallation target and contains high  $^{99}\text{Tc}$  activity. Because of this,  $^{95\text{m}}\text{Tc}$  cannot be added to every sample and instead splits of samples are taken for recovery measurements. Future work will employ  $^{95\text{m}}\text{Tc}$  which is produced by alpha bombardment of  $^{93}\text{Nb}$ , which is reported to produce  $^{99}\text{Tc}$ -free material. Results of the tracer work show typically high recovery (> 90%) for aqueous samples and mixed results for soil leachates, depending on the soil type. Transfers of pertechnetate onto and off of the column, and ultimately to the final sample pellet, are consistently quantitative.

### ***AMS Measurement***

AMS measurements are performed using the 10 MV FN tandem accelerator at LLNL (Southon, Caffee et al. 1990). The setup used for  $^{99}\text{Tc}$  measurements is shown in Figure 1.  $^{93}\text{Nb}^{16}\text{O}^-$  and  $^{99}\text{Tc}^{16}\text{O}^-$  ions are accelerated to 40 kV and quasi-simultaneously injected into the accelerator. The injection cycle is ~3Hz (1 ms  $^{93}\text{Nb}^{16}\text{O}^-$ ; 300 ms  $^{99}\text{Tc}^{16}\text{O}^-$ ). The accelerating voltage is 9.0 MV and foil stripping is used. The high energy spectrometer is set to select  $^{99}\text{Tc}^{13+}$  ions at 125 MeV energy. Extracted  $^{93}\text{Nb}^{16}\text{O}^-$  currents are typically 1-2  $\mu\text{A}$ , and stripping yield in the 13+ charge state is 4-5%.  $^{93}\text{Nb}^{13+}$  current is measured in an off-axis Faraday cup located after the first high energy analyzing magnet.

Ion counting is performed using a gas ionization detector filled to 110 Torr with P10 (90% argon, 10% methane). Differential energy loss is measured in 3 anodes. Two dimensional spectra (first anode v. third anode), gated on the second anode, are used for particle identification.

Ruthenium and technetium peaks are not fully resolved at the energies available with the LLNL tandem. Windows are drawn to select Tc and Ru respectively. The Tc window is drawn to select ~85% of the Tc events, and ~25% of the Ru events. For quality control, a third window is included to monitor for Mo events which are occasionally evident in the spectra, however for environmental samples the detected Mo rate is nearly always negligible compared to the Ru rate. Corrected Tc, Ru, and Mo counts are determined analytically from the gated events. Counting uncertainties and systematic uncertainties in the deconvolution (caused by small gain shifts in the detector) are included in the corrected results. The parameters (the fractions of each type of event in each cut) for the deconvolution are determined by repeatedly running blanks, high activity Tc standards, and spiked Ru samples as part of the measurement protocol.

Ratios of Tc counts to Nb integrated charge, corrected for electronics deadtime and  $^{99}\text{Ru}$  background, are computed for each sample. This ratio is converted total sample  $^{99}\text{Tc}$  by

normalizing to the same ratio as measured for  $^{99}\text{Tc}$  standards, then multiplying by the amount of Nb carrier solution added.

### ***Technetium-99 Results***

To date we have measured  $^{99}\text{Tc}$  in soil leachates and fresh and sea water samples. Sensitivity of 10-20 fg is typical for each sample type, and low-level standards containing 20 fg  $^{99}\text{Tc}$  are readily measureable above background. Three difficulties have been encountered in these measurements. These are: variability in the measured ratios; matrix material which is not removed during sample preparation; and relatively high ruthenium levels in some samples.

To evaluate our methods, we have performed measurements of the reference material IAEA-381, Irish Sea water. A  $^{99}\text{Tc}$  activity of  $217 \pm 11$  mBq/kg was reported for this material in (Povinec, Phan et al. 1999). Figure 2 shows the results for a set of 20 IAEA-381 aliquots which were included as part of a set of ~120 environmental samples measured over a 3 day period. The average of the AMS results is 190 mBq/kg, with a standard deviation of  $\pm 14\%$ . No correction for chemical recovery is included in these results. If the two most extreme outliers are removed, the average remains 190 mBq/kg and the standard deviation drops to  $\pm 10\%$ .

The variability observed in the IAEA-381 results is typical of that typically seen in samples and standards. The source of this sample-to-sample variation is time-dependence in the Tc/Nb ratio measured for an individual sample over the course of the measurement. This time-dependence has also been reported by the AMS group at the Australian National University (in that case Tc/Rh ratios are measured) (Fifield 1998). It is thought that this is caused by differences in the sputtering rates of Tc and Nb, though sample inhomogeneity may also play a role. The effect of this time-dependence can be reduced by integrating the measurement over a longer time, and assuring that the integration time is as similar as possible for different samples. However, at present this represents the major limitation to our measurements.

Fresh water samples from 1-200 mL have been measured with ~15 fg sensitivity. Based on the relative ease of sample preparation for water samples, samples as large as 1 L should be easily measureable with an expected sensitivity of ~0.01 mBq/kg (~0.3 fCi/L).

Soil leachates have been measured in arid soils for samples up to 250 g. A difficulty in the large sample (and in some ~100 mL freshwater samples) is the presence of significant quantities (milligrams) of non-volatile material (presumably soluble salts) which are not fully removed by the sample chemistry. For quality control, each pellet is weighed after baking. The primary effect of this material is dilution of the niobium and technetium, resulting in low or unmeasurable signals. Since it is not known how this dilution might affect the measured Tc/Nb ratio, these samples are flagged as suspect. Efforts to fully identify and remove this remaining material is ongoing. Initial experience shows that precipitation steps using calcium and barium, combined with additional rinse volumes of the column prior to elution, can reduce this material to acceptable levels in most cases.

In a recent depth profile of an arid soil using 60-220 g aliquots, the average and standard deviation of 18 baseline samples was  $1 \pm 11$  fg per aliquot. Surface activity, presumably from fallout, of 0.1-2 mBq/kg was readily measurable above background. These results are still preliminary, but they demonstrate the sensitivity of the method.

## **3. Plutonium measurements**

### ***Technical Issues***

Initial emphasis for actinide AMS at LLNL is being placed on measurements of  $^{239,240}\text{Pu}$  concentrations in urine. Target sensitivity for this application is ~1  $\mu\text{Bq/L}$  (~ $1 \times 10^6$  atoms per sample). The overriding technical issue for achieving this sensitivity is rejection of interference at mass 239 from  $^{238}\text{U}$ . A series of hardware upgrades to the CAMS spectrometer are required, which include a heavy ion beamline (see Figure 3) capable of transporting these heavy ions, and a large electrostatic analyzer (ESA) to improve rejection of interference from  $^{238}\text{U}$  (McAninch

and Hamilton 1999). Work to date has been performed on a prototype beamline which does not include the ESA, but instead has a Wien filter for limited velocity selection.

Other technical issues surrounding this development were selection of sample matrix, chemical removal of sample uranium, level of uranium background in the sample matrix and ion source, negative ion rate and efficiency, optimum accelerator parameters and stripping yield. Much of the basic work on these issues was performed by Fifield, *et al.* (Fifield, Cresswell *et al.* 1996; Fifield, Clacher *et al.* 1997).

### ***Sample Preparation***

For the soil samples reported here, the initial sample preparation followed the standard protocols used for alpha spectrometry at HEA. This procedure results in a final column eluant, from which the Pu is electrodeposited on a planchet. For these samples, prior to the electrodeposition step, five 10% splits were taken for AMS and the remainder was kept for alpha counting.

The AMS aliquots (in HCl) were taken to dryness, then re-dissolved in high purity nitric acid. Iron (1 mg Fe as iron nitrate in atomic absorption standard solution) was added, and the sample was again taken to dryness, then baked to 800° C. The resulting oxide is then mixed with 1 mg of high purity niobium powder (-325 mesh), then pressed into a standard LLNL aluminum AMS sample holder. Two of the 5 splits were measured by AMS, and the rest were preserved for future comparison measurements.

### ***AMS Measurement***

$^{238}\text{U}^{16}\text{O}^-$  and  $^{239,240,241,242}\text{Pu}^{16}\text{O}^-$  ions are accelerated to 28 kV and sequentially injected into the accelerator. The accelerating voltage is 6.5 MV and gas stripping is used. The high energy spectrometer is set to select 5+ ions at 39 MeV energy. The injection line, 30° high energy analyzing magnet, and Wien filter are changed for each mass selection.

Ion counting is performed using a 2-anode, longitudinal field gas ionization detector filled to 90 Torr with P10 (90% argon, 10% methane). Differential energy loss and total energy is used for particle identification. Interfering ions at 4+ and lower charge states are cleanly resolved by the detector.

The data reported here consist of a single pass through each sample (242, 238, 239, 240, 241, 242). Following warm-up of samples and initial measurements, each mass was measured for 30 s, with up to 30 s switching time between masses, caused by the long response time of the 30° analyzing magnet. Count rates at each mass, corrected for electronics deadtime, were normalized to the average 242 rate for that sample, then multiplied by the known  $^{242}\text{Pu}$  spike activity. No corrections for uranium or other backgrounds were employed.

With the prototype beamline, backgrounds at masses 237 and 239 are  $\sim 1\text{--}2 \times 10^7$  atoms per sample, limited by  $^{238}\text{U}$  interference and possible Pu introduced during sample preparation. Backgrounds at masses 240 and 241 are  $< 5 \times 10^6$  atoms per sample, limited by counting efficiency for 30 s counts.

Rejection of  $^{238}\text{U}$  is expected to be improved by a factor of 10-100 with the electrostatic analyzer and additional hardware modifications. This should reduce instrumental backgrounds to  $< 1 \times 10^6$  atoms per sample at masses 237, 239, 240, and 241, and should also significantly reduce the demands for chemical removal of uranium during sample preparation.

### ***Plutonium Results***

Results for AMS and alpha spectrometry measurements of Pu in IAEA reference materials are shown in Tables 1 and 2. The results are compared to IAEA reference values and to recent measurements of two of these samples by ICP-MS reported by Muramatsu, *et al.* (Muramatsu, Uchida *et al.* 1999).

The  $^{239+240}\text{Pu}$  activities as measured by AMS (Table 1) show a significant bias with increasing activity (from +20% to -20%) relative to the IAEA reference values. However, the same bias is

seen in the alpha spectrometry measurements of the sample splits, indicating that the bias results from incomplete equilibration of the  $^{242}\text{Pu}$  spike and sample Pu.

The precision/accuracy of the AMS results (as seen in the differences between separate AMS aliquots) is limited by time-dependence in the sample output during the measurements. This time-dependence is not averaged out using the slow-switching mode and single pass measurement employed in this data set. A fast switching mode and multiple passes for each sample will be used with the final implementation of the heavy element beamline.

The AMS results for  $^{240}\text{Pu}/^{239}\text{Pu}$  isotope ratios in IAEA-135 showed good agreement to the published ICP-MS results. Agreement (within the AMS uncertainties) was also obtained for IAEA Soil 6.  $^{241}\text{Pu}$  activities were also measured in the samples to examine our capabilities for other isotopes.

### 3. Conclusions

We have demonstrated the feasibility of measurements of  $^{99}\text{Tc}$  and  $^{239,240,241}\text{Pu}$  by AMS using the LLNL spectrometer. Instrumental development for  $^{99}\text{Tc}$  measurements is complete, and measurements for some sample matrices are now considered to be routine. Final development of capability for Pu and other actinides is dependent on the hardware upgrades, which are expected to be complete in the next few months.

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**Table 1. Pu-239+240 activity concentrations (PRELIMINARY).** Recent measurements of  $^{239}\text{Pu}+^{240}\text{Pu}$  activity concentrations (in Bq/kg) in IAEA reference materials by accelerator mass spectrometry (AMS) and alpha spectrometry at Lawrence Livermore National Laboratory. The results are compared to IAEA reference values. Following sample digestion and purification, splits of the sample were taken for alpha spectrometry (1 @ 50%) and AMS analysis (5 @ 10%, of which 2 are shown below). Precision/accuracy for the AMS results is presently limited by slow switching of the spectrometer between masses. This will be significantly improved with planned hardware and software upgrades, and increase of data acquisition times to 3-5 min per isotope.

IAEA Sample	IAEA reference value	AMS result <sup>1</sup> (Bq/kg)	$\alpha$ -spec result (Bq/kg)	AMS / ref %	$\alpha$ -spec / ref %	Sample Size, g
<b>IAEA-135</b> (Irish Sea Sediment)	213	244 $\pm$ 2 246 $\pm$ 2	252	115 % 115 %	118 %	1
<b>IAEA-367</b> (Pacific Ocean Sediment)	38	36.6 $\pm$ 0.2 31.9 $\pm$ 0.2	38.5	96 % 84 %	101 %	10
<b>IAEA, Soil 6</b> (Austrian Soil)	1.04	0.96 $\pm$ 0.04 0.92 $\pm$ 0.04	0.97	92 % 88 %	94 %	10
<b>IAEA-375</b> (Chernobyl Soil)	0.30	0.27 $\pm$ 0.02 0.21 $\pm$ 0.02	0.23	91 % 70 %	77 %	10
<b>Reagent Blank</b> (10 g equivalent)	—	0.014 $\pm$ 0.006 0.014 $\pm$ 0.007 (values not corrected for $^{238}\text{U}$ interference)	<0.1	— —	— —	— —

<sup>1</sup> The AMS results have not been corrected for U-238 interference. Quoted AMS uncertainties include counting statistics only.

**Table 2. Pu-240/239 isotope ratios and Pu-241 activity concentrations (PRELIMINARY).** Recent measurements of plutonium isotopes in IAEA reference materials by accelerator mass spectrometry (AMS) and alpha spectrometry at Lawrence Livermore National Laboratory. The results are compared to recently published results obtained using ICP-MS.<sup>1</sup> For further information see Table 1.

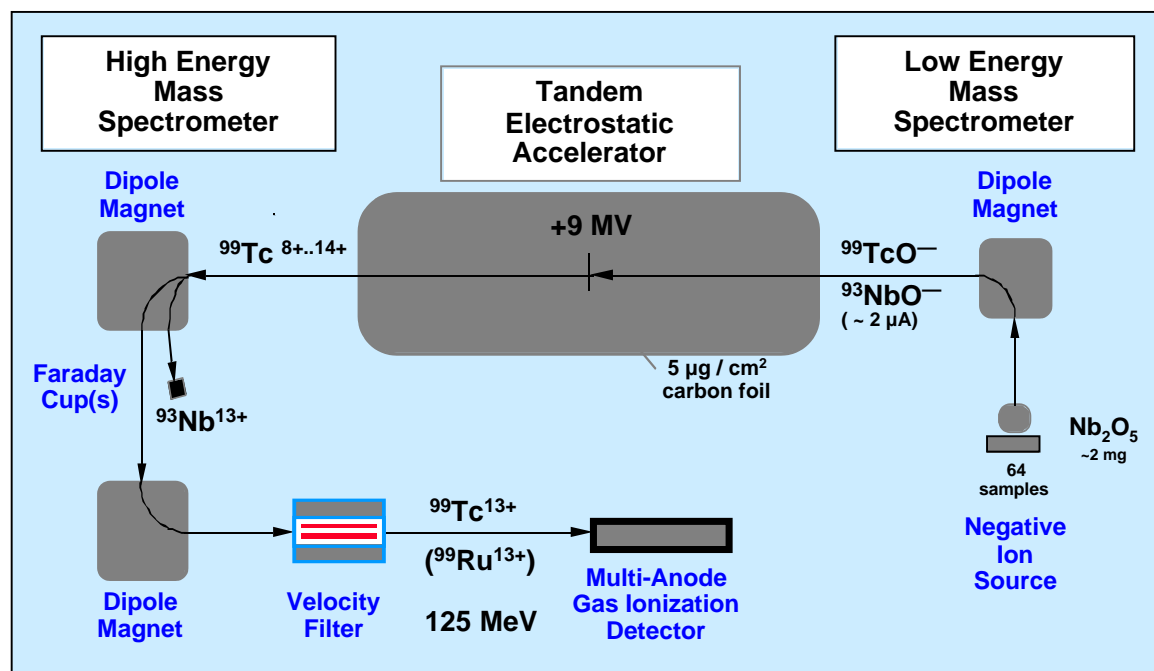
IAEA Sample	240/239 ICP-MS result <sup>1</sup> %	240/239 AMS result <sup>2</sup> %	241 activity AMS result <sup>2,3</sup> Bq/kg
<b>IAEA-135</b> (Irish Sea Sediment)	21.1 ± 0.4	21.3 ± 0.6 21.1 ± 0.6	2910 ± 180 2940 ± 200
<b>IAEA, Soil 6</b> (Austrian Soil)	19.1 ± 0.5	17 ± 4 18 ± 4	1 ± 2 6 ± 3
<b>IAEA-367</b> (Pacific Ocean Sediment)	—	32.0 ± 0.6 29.6 ± 0.5	116 ± 10 <sup>4</sup> 90 ± 8 <sup>4</sup>

<sup>1</sup> ICP-MS results from Muramatsu, Y., S. Uchida, et al. (1999). "Determination of plutonium concentration and its isotopic ratio in environmental materials by ICP-MS after separation using ion-exchange and extraction chromatography." *Journal of Analytical and Atomic Spectroscopy* **14**: 859-865..

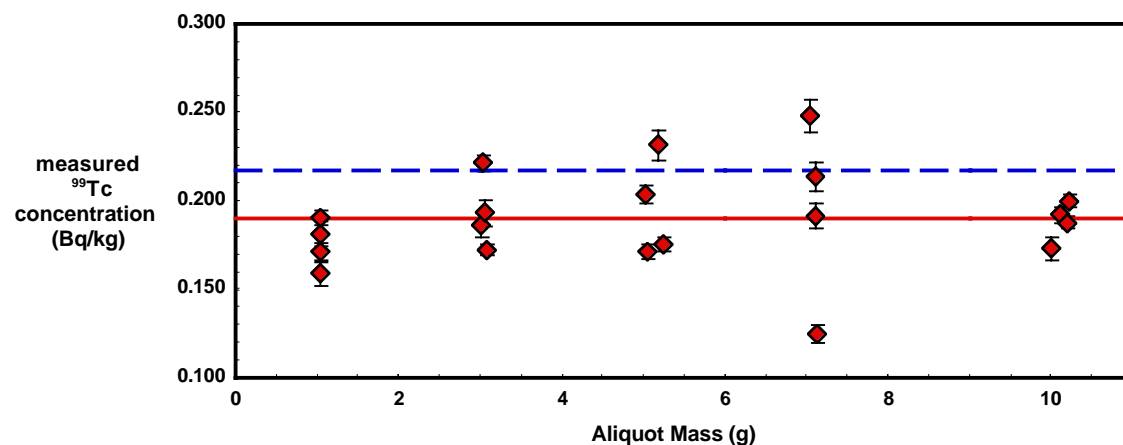
<sup>2</sup> The AMS results have not been corrected for U-238 interference. Quoted AMS uncertainties include counting statistics only.

<sup>3</sup> Pu-241 activity on 8-AUG-1999.

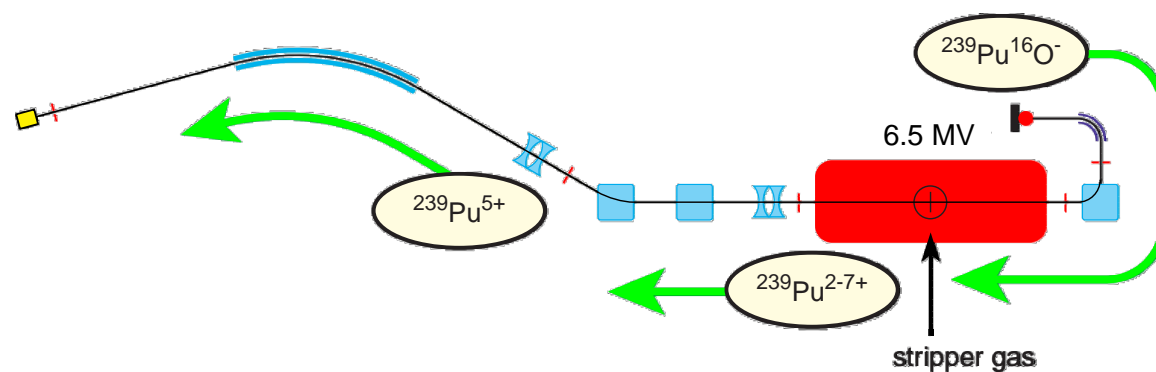
<sup>4</sup> The IAEA provides an information value for Pu-241 activity in this sample of 170 Bq/kg referenced to 1-JAN-1990. Decayed to 8-AUG-1999 this information value becomes 107 Bq/kg.



**Figure 1. Accelerator mass spectrometry measurements of  $^{99}\text{Tc}$  at LLNL.** Samples are prepared as technetium oxide in a niobium oxide matrix, then placed in the Cs-sputter source. The resulting  $^{99}\text{Tc}^{16}\text{O}^-$  and  $^{93}\text{Nb}^{16}\text{O}^-$  ions are accelerated to 9 MV, then passed through a thin carbon foil where the molecules are dissociated and outer electrons are stripped. The high energy spectrometer is set to accept  $^{99}\text{Tc}^{13+}$  ions, which are transported to the gas ionization detector following rigidity and velocity analysis.  $^{93}\text{Nb}^{13+}$  currents are measured in an off-axis Faraday cup following the first high energy magnet.



**Figure 2. Accelerator mass spectrometry measurements of <sup>99</sup>Tc in IAEA-381 Irish Sea Water (PRELIMINARY).** A <sup>99</sup>Tc concentration in IAEA-381 of 217±11 mBq/kg (dashed line) was reported in (Povinec, Phan et al. 1999). For the present work, 20 aliquots of different masses were prepared as described in the text, then analyzed with other samples over the course of 3 days. The average for all values (solid line) is 190 mBq/kg with a standard deviation of ±14%. When the two most extreme outliers (both were 7 g samples), the average remains 190 mBq/kg, and the standard deviation drops to ±10%. Given the large scatter of the AMS results, and the still developmental status of the methods, these AMS results should be considered preliminary. In addition, no <sup>95m</sup>Tc recovery measurements were performed for this material.



**Figure 3. Accelerator mass spectrometry measurements of Pu isotopes at LLNL: Planned heavy element beamline.** Samples are prepared as plutonium oxide in an iron oxide matrix, then placed in the Cs-sputter source.  $^{239-242}\text{Pu}^{16}\text{O}^-$  are successively accelerated to 6.5 MV, then passed through a thin gas stripping region where the molecules are dissociated and outer electrons are stripped. The high energy spectrometer is set to successively accept  $^{239-242}\text{Pu}^{5+}$  ions, which are transported to the gas ionization detector following rigidity and energy analysis. A large electrostatic analyzer (4.4 m radius,  $45^\circ$  bending angle, 50 kV/cm maximum field) will provide the final separation of interferences. For the work reported here, a prototype version of the heavy element beamline was used. In the prototype, a Wein velocity filter was used in place of the electrostatic analyzer.